

DRAFT CLAIMS — DO NOT ENTER

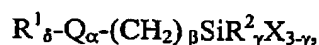
Docket No.: 03-34US
Serial No. 10/777,523

DRAFT CLAIMS FOR DISCUSSION WITH EXAMINER THERKORN

In the Claims:

This listing of claims will replace all prior versions, and listings, of claims in the application:

1. (previously presented) A composition for use as a stationary phase in chromatography comprising an inorganic substrate that is modified with at least one silane having the formula



wherein R^1 is hydrogen, $C_1 - C_{100}$ substituted or unsubstituted hydrocarbyl, cycloalkyl, heterocycloalkyl, aryl, or heteroaryl; wherein the substituents are selected from $C_1 - C_{12}$ hydrocarbyl, hydroxyl, alkoxy, halogen, amino, nitro, sulfo, and carbonyl;

α is 0 or 1;

β is 0-30;

γ is 0, 1 or 2;

δ is 0-3;

R^2 is $C_1 - C_{100}$ substituted or unsubstituted hydrocarbyl, cycloalkyl, heterocycloalkyl, aryl, or heteroaryl; wherein the substituents are selected from $C_1 - C_{12}$ hydrocarbyl, hydroxyl, alkoxy, halogen, amino, nitro, sulfo, and carbonyl;

Q is independently selected from -NHC(O)-, -C(O)NH-, -OC(O)NH-, -NHC(O)O-, -NHC(O)NH-, -NCO-, -CHOHCHOH-, -CH₂OCHCH₂O-, -(CH₂CH₂O)_n-, -(CH₂CH₂CH₂O)_n-, -C(O)-, -C(O)O-, -OC(O)-, CH₃C(O)CH₂-, -S-, -SS-, -CHOH-, -O-, -SO-, -SO₂-, -SO₃-, -OSO₃-, -SO₂NH-, -SO₂NMe-, -NH-, -NMe-, -NMe₂⁺-, -N[(CH₂)_n]₂⁺-, -CN, -NC, -CHOCH-, -NHC(NH)NH-, -NO₂-, -NO-, -OPO₃-, where n is 1-30; and

X is a leaving group; and

wherein said inorganic substrate is equilibrated in an atmosphere having a defined relative humidity of less than 50% prior to being modified with the at least one silane.

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2. (original) The composition of claim 1, wherein the inorganic substrate is a metal oxide or metalloid oxide.
3. (original) The composition of claim 2, wherein the inorganic substrate is in the form of a monolith or porous particles.
4. (original) The composition of claim 2, wherein the inorganic substrate comprises silica.
5. (original) The composition of claim 3, wherein said porous particles have an average pore diameter from about 60 Å to about 1000 Å.
6. (original) The composition of claim 3, wherein said porous particles have an average particle size from about 3 µm to about 60 µm.
7. (canceled) The composition of claim 1, wherein said inorganic substrate is equilibrated in an atmosphere having a defined relative humidity prior to being modified with the at least one silane.
8. (original) The composition of claim 1 7, wherein the atmosphere having a defined relative humidity is provided by hydrated salts or saturated salt solutions.
9. (original) The composition of claim 4, wherein the inorganic substrate comprises silica gel modified with at least two silanes.
10. (original) The composition of claim 9, wherein the silica gel is modified with a first silane, and subsequently the silica gel substrate is modified with a second silane.

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11. (original) The composition of claim 10, wherein the first or second silane or both the first and the second silanes comprises a mixture of silanes.
12. (original) The composition of claim 9, wherein the silica gel substrate is modified with at least one silane wherein δ is from 0 to 3, and at least one additional silane wherein δ is 0 or 1.
13. (original) The composition of claim 9, wherein the additional silane is an endcapping silane.
14. (original) The composition of claim 13 9, wherein the endcapping silane is a monosilane, disilane, trisilane or tetrasilane, or a combination thereof.
15. (original) The composition of claim 14, wherein the monosilane is trimethylchlorosilane, N,N-dimethyltrimethylsilylamine, trimethylsilylimidazole, dimethyldichlorosilane, dimethoxydimethylsilane, trimethylsilanol, trimethylsilylphosphine, or N-trimethylsilylacetamide.
16. (original) The composition of claim 14, wherein the disilane is hexamethyldisilazane or 1,3-dimethoxytetramethyldisiloxane.
17. (original) The composition of claim 14, wherein the trisilane is hexamethylcyclotrisiloxane.
18. (original) The composition of claim 14, wherein the tetrasilane is octamethylcyclotetrasiloxane.
19. (original) The composition of claim 1, wherein X is halogen, alkoxy, amino, or acyloxy.

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20. (original) The composition of claim 1, wherein Q, R¹ or R² is a chiral recognition ligand.

21. (original) The composition of claim 20, wherein the chiral recognition ligand is optically active.

22. (original) The composition of claim 20, wherein the chiral recognition ligand is a cyclodextrin.

23. (original) The composition of claim 9, wherein the silica gel is modified by the following steps:

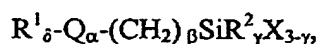
- (a) equilibrating the silica gel in an atmosphere having a defined relative humidity;
- (b) modifying the silica gel with at least one silane; and
- (c) further modifying the silica gel with an endcapping silane.

24. (original) The composition of claim 23, further comprising the step of modifying the silica gel with a second silane.

25. (withdrawn) A method for modifying an inorganic substrate, comprising the steps of:

- (a) equilibrating the inorganic substrate in an atmosphere having a defined relative humidity to provide a controlled amount of water on the surface of the inorganic substrate;
- (b) modifying the inorganic substrate with at least one silane; and
- (c) further modifying the inorganic substrate with an endcapping silane.

26. (withdrawn) The method of claim 25, wherein the silane has the formula:



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wherein R^1 is hydrogen, $C_1 - C_{100}$ substituted or unsubstituted hydrocarbyl, cycloalkyl, heterocycloalkyl, aryl, or heteroaryl; wherein the substituents are selected from $C_1 - C_{12}$ hydrocarbyl, hydroxyl, alkoxy, halogen, amino, nitro, sulfo, and carbonyl;

α is 0 or 1;

β is 0-30;

γ is 0, 1 or 2;

δ is 0-3;

R^2 is $C_1 - C_{100}$ substituted or unsubstituted hydrocarbyl, cycloalkyl, heterocycloalkyl, aryl, or heteroaryl; wherein the substituents are selected from $C_1 - C_{12}$ hydrocarbyl, hydroxyl, alkoxy, halogen, amino, nitro, sulfo, and carbonyl;

Q is independently selected from -NHC(O)-, -C(O)NH-, -OC(O)NH-, -NHC(O)O-, -NHC(O)NH-, -NCO-, -CHOHCHOH-, -CH₂OCHCH₂O-, -(CH₂CH₂O)_n-, -(CH₂CH₂CH₂O)_n-, -C(O)-, -C(O)O-, -OC(O)-, CH₃C(O)CH₂-, -S-, -SS-, -CHOH-, -O-, -SO-, -SO₂-, -SO₃-, -OSO₃-, -SO₂NH-, -SO₂NMe-, -NH-, -NMe-, -NMe₂⁺-, -N[(CH₂)_n]₂⁺-, -CN-, -NC-, -CHOCH-, -NHC(NH)NH-, -NO₂-, -NO-, -OPO₃-, where n is 1-30; and

X is a leaving group.

27. (withdrawn) The method of claim 26, further comprising the step of modifying the inorganic substrate with a second silane, wherein δ for the second silane is from 0-3.

28. (withdrawn) The method of claim 27, wherein the modification step with the second silane is performed at the same time as the modification step with the first silane.

29. (withdrawn) The method of claim 27, wherein the modification step with the second silane is performed after the modification step with the first silane.

30. (withdrawn) The method of claim 25, wherein the atmosphere having a defined relative humidity is provided by hydrated salts or saturated salt solutions.

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31. (withdrawn) The method of claim 30, wherein the defined relative humidity is less than 50%.
32. (withdrawn) The method of claim 30, wherein the relative humidity is from about 0% to about 10%, from about 10% to about 20%, from about 20% to about 30%, from about 40% to about 50%, from about 50% to about 60%, from about 60% to about 70%, from about 70% to about 80%, from about 80% to about 90% or from about 90% to about 100%.
33. (withdrawn) The method of claim 30, wherein the hydrated salts or saturated salt solutions comprise cesium fluoride, lithium bromide, zinc bromide, potassium hydroxide, sodium hydroxide, lithium chloride, calcium bromide, potassium acetate, potassium fluoride, magnesium chloride, sodium iodide, potassium carbonate, magnesium nitrate, sodium bromide, cobalt chloride, sodium nitrite, potassium iodide, strontium chloride, sodium nitrate, sodium chloride, ammonium chloride, potassium bromide, ammonium sulfate, potassium chloride, strontium nitrate, barium chloride, potassium nitrate, or potassium sulfate.
34. (withdrawn) The method of claim 25, wherein the inorganic substrate is a metal or metalloid oxide substrate.
35. (withdrawn) The method of claim 34, wherein the metal or metalloid oxide comprises silica, alumina, zeolite, mullite, zirconia, vanadia or titania, or mixtures thereof.
36. (withdrawn) A method for separating a plurality of analytes, comprising performing a chromatographic separation using a stationary phase comprising an inorganic substrate modified by at least one silane according to claim 1.

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37. (withdrawn) The method of claim 36, wherein the chromatographic separation is performed using a gaseous or liquid mobile phase.

38. (withdrawn) The method of claim 37, wherein the mobile phase comprises from 0 to 100% water.

39. (withdrawn) The method of claim 37, wherein the chromatographic separation is performed using thin layer chromatography, high performance liquid chromatography, reversed phase chromatography, normal phase chromatography, ion chromatography, ion pair chromatography, reverse phase ion pair chromatography, ion exchange chromatography, affinity chromatography, hydrophobic interaction chromatography, size exclusion chromatography, chiral recognition chromatography, perfusion chromatography, electrochromatography, partition chromatography, microcolumn liquid chromatography, capillary chromatography, liquid-solid chromatography, preparative chromatography, hydrophilic interaction chromatography, supercritical fluid chromatography, precipitation liquid chromatography, bonded phase chromatography, fast liquid chromatography, flash chromatography, liquid chromatography mass spectrometry, gas chromatography, microfluidics based separations, solid phase extraction separations, or monolith based separations.

40. (original) The composition of claim 1, wherein the inorganic substrate is modified by the following steps:

- a) equilibrating the inorganic substrate in an atmosphere having a defined relative humidity;
- b) modifying the inorganic substrate with at least one silane; and
- c) further modifying the inorganic substrate with an endcapping silane.

41. (original) The composition of claim 1, wherein the retention time, peak symmetry and retention factor for analytes separated on said stationary phase varies by no more than about 5% even when exposed to acidic or basic elution conditions for 3000 hours.

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42. (original) The composition of claim 10, wherein the first silane has a value for δ of 1, α is 1, β is 1-30, γ is 0, 1, or 2, R^1 is a substituted or unsubstituted $C_1 - C_{30}$ hydrocarbyl, Q is amido or carbamyl, and the second silane has a value for δ of 1, α is 1, β is 1-30, γ is 0, 1, or 2, R^1 is a substituted or unsubstituted $C_1 - C_6$ hydrocarbyl, and Q is amido, carbamyl, cyano or glycidoxo.

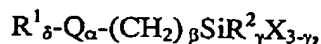
43. (original) The composition of claim 10, wherein the first silane has a value for δ of 1, α is 0, β is 8-30, γ is 0, 1 or 2, R^1 is H, and the second silane has a value for δ of 1, R^1 is a substituted or unsubstituted $C_1 - C_6$ hydrocarbyl, and Q is amido, cyano or glycidoxo.

44. (original) The composition of claim 10, wherein the first silane has a value for δ of 1, α is 0, β is 8-30, γ is 0, 1 or 2, R^1 is H, and the second silane has a value for δ of 1, R^1 is a substituted or unsubstituted $C_1 - C_6$ hydrocarbyl, and Q is thio, dithio, ether, sulfinyl, sulfonyl, sulfonic acid, sulfate, sulfonamido, amino, nitrile, isonitrile, epoxy, guanidino, nitro, nitroso, or phosphate.

45. (presently amended) The composition of claim 1, wherein the relative humidity is from about 0% to about 10%, from about 10% to about 20%, from about 20% to about 30%, from about 30% to about 40%, or from about 40% to about 50%.

Please add the following new claims:

46. (new) A composition for use as a stationary phase in chromatography comprising an inorganic substrate that is modified with at least two silanes having the formula



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wherein R^1 is hydrogen, $C_1 - C_{100}$ substituted or unsubstituted hydrocarbyl, cycloalkyl, heterocycloalkyl, aryl, or heteroaryl; wherein the substituents are selected from $C_1 - C_{12}$ hydrocarbyl, hydroxyl, alkoxy, halogen, amino, nitro, sulfo, and carbonyl;

α is 0 or 1;

β is 0-30;

γ is 0, 1 or 2;

δ is 0-3;

R^2 is $C_1 - C_{100}$ substituted or unsubstituted hydrocarbyl, cycloalkyl, heterocycloalkyl, aryl, or heteroaryl; wherein the substituents are selected from $C_1 - C_{12}$ hydrocarbyl, hydroxyl, alkoxy, halogen, amino, nitro, sulfo, and carbonyl;

Q is independently selected from -NHC(O)-, -C(O)NH-, -OC(O)NH-, -NHC(O)O-, -NHC(O)NH-, -NCO, -CHOHCHOH-, -CH₂OCHCH₂O-, -(CH₂CH₂O)_n-, -(CH₂CH₂CH₂O)_n-, -C(O)-, -C(O)O-, -OC(O)-, CH₃C(O)CH₂-, -S-, -SS-, -CHOH-, -O-, -SO-, -SO₂-, -SO₃-, -OSO₃-, -SO₂NH-, -SO₂NMe-, -NH-, -NMe-, -NMe₂+, -N[(CH₂)_n]²⁺-, -CN, -NC, -CHOCH-, -NHC(NH)NH-, -NO₂, -NO, -OPO₃-, where n is 1-30; and

X is a leaving group.

47. (new) The composition of claim 46, wherein the inorganic substrate is a metal oxide or metalloid oxide.

48. (new) The composition of claim 47, wherein the inorganic substrate is in the form of a monolith or porous particles.

49. (new) The composition of claim 47, wherein the inorganic substrate comprises silica.

50. (new) The composition of claim 48, wherein said porous particles have an average pore diameter from about 60 Å to about 1000 Å.

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51. (new) The composition of claim 3, wherein said porous particles have an average particle size from about 3 μm to about 60 μm .

52. (new) The composition of claim 1, wherein said inorganic substrate is equilibrated in an atmosphere having a defined relative humidity prior to being modified with the at least one silane.

53. (new) The composition of claim 52, wherein the atmosphere having a defined relative humidity is provided by hydrated salts or saturated salt solutions.

54. (new) The composition of claim 46, wherein the silica gel is modified with a first silane, and subsequently the silica gel substrate is modified with a second silane.

55. (new) The composition of claim 54, wherein the first or second silane or both the first and the second silanes comprises a mixture of silanes.

56. (new) The composition of claim 46, wherein the silica gel substrate is modified with at least one silane wherein δ is from 0 to 3, and at least one additional silane wherein δ is 0 or 1.

57. (new) The composition of claim 54, wherein the first silane has a value for δ of 1, α is 1, β is 1-30, γ is 0, 1, or 2, R^1 is a substituted or unsubstituted $C_1 - C_{30}$ hydrocarbyl, Q is amido or carbamyl, and the second silane has a value for δ of 1, α is 1, β is 1-30, γ is 0, 1, or 2, R^1 is a substituted or unsubstituted $C_1 - C_6$ hydrocarbyl, and Q is amido, carbamyl, cyano or glycidoxo.

58. (new) The composition of claim 54, wherein the first silane has a value for δ of 1, α is 0, β is 8-30, γ is 0, 1 or 2, R^1 is H, and the second silane has a value for δ of 1, R^1 is a substituted or unsubstituted $C_1 - C_6$ hydrocarbyl, and Q is amido, cyano or glycidoxo.

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59. (new) The composition of claim 54, wherein the first silane has a value for δ of 1, α is 0, β is 8-30, γ is 0, 1 or 2, R^1 is H, and the second silane has a value for δ of 1, R^1 is a substituted or unsubstituted $C_1 - C_6$ hydrocarbyl, and Q is thio, dithio, ether, sulfinyl, sulfonyl, sulfonic acid, sulfate, sulfonamido, amino, nitrile, isonitrile, epoxy, guanidino, nitro, nitroso, or phosphate.

60. (new) The composition of claim 46, wherein the inorganic substrate is further modified with an endcapping silane.

61. (new) The composition of claim 60, wherein the endcapping silane is a monosilane, disilane, trisilane or tetrasilane, or a combination thereof.

62. (new) The composition of claim 46, wherein X is halogen, alkoxy, amino, or acyloxy.

63. (new) The composition of claim 46, wherein Q, R^1 or R^2 is a chiral recognition ligand.

64. (new) The composition of claim 63, wherein the chiral recognition ligand is optically active.

65. (new) The composition of claim 63, wherein the chiral recognition ligand is a cyclodextrin.

66. (new) The composition of claim 46, wherein the silica gel is modified by the following steps:

- (d) equilibrating the silica gel in an atmosphere having a defined relative humidity;
- (e) modifying the silica gel with at least two silanes; and
- (f) further modifying the silica gel with an endcapping silane.